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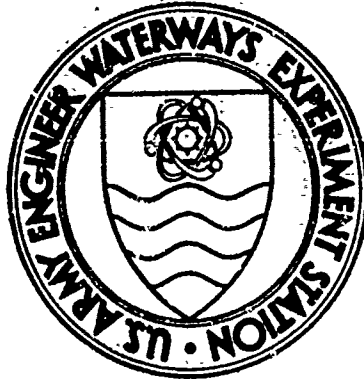
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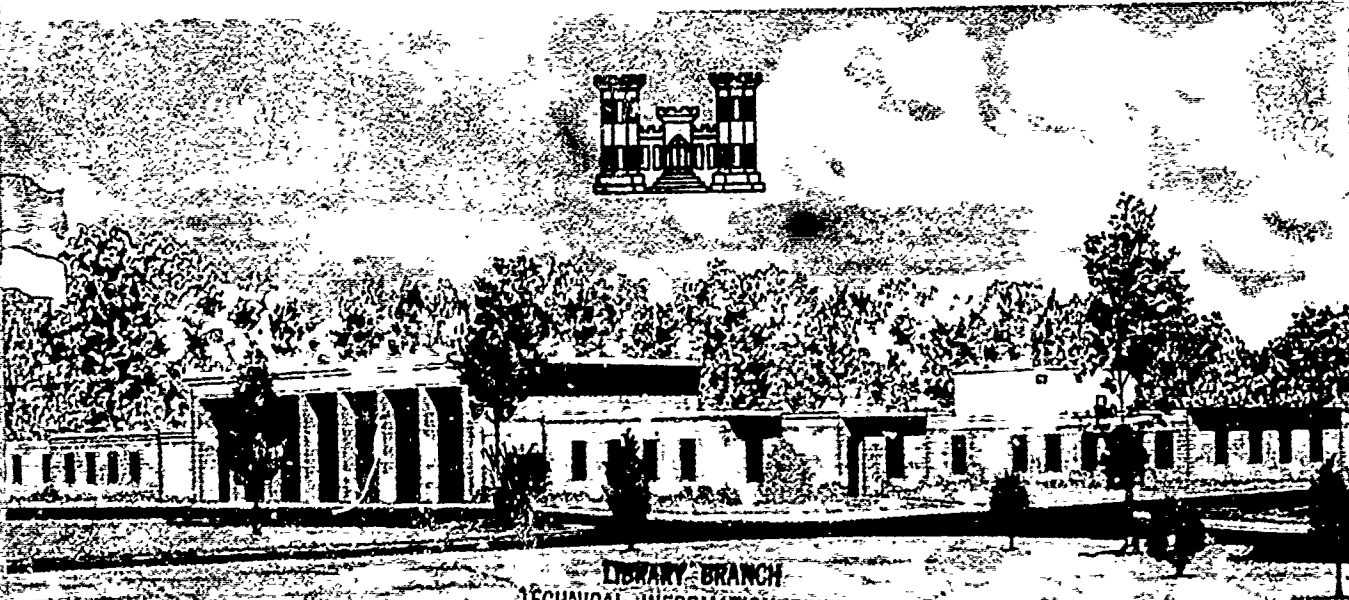


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# INVESTIGATION OF ACCELERATED METHODS FOR THE DETERMINATION OF AVAILABLE ALKALI IN POZZOLANS

W. G. Miller



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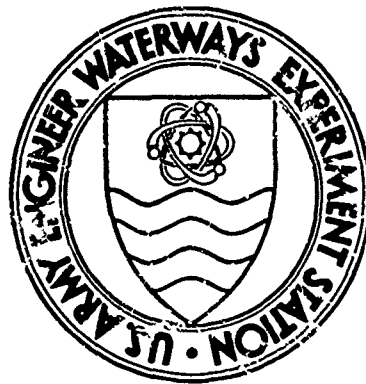
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|     | Acceptance tests     |        |    |        |    |        |    |
|     | Alkalies             |        |    |        |    |        |    |
|     | Lime-pozzolan pastes |        |    |        |    |        |    |
|     | Pozzolans            |        |    |        |    |        |    |



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# INVESTIGATION OF ACCELERATED METHODS FOR THE DETERMINATION OF AVAILABLE ALKALI IN POZZOLANS

by

W. G. Miller



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### Foreword

The investigation reported herein was authorized by the Office, Chief of Engineers (OCE), by the first indorsement, dated 30 March 1970, to a letter from OCE to the U. S. Army Engineer Waterways Experiment Station (WES), dated 25 February 1970, subject: "Project Plan for Investigation of Accelerated Methods for the Determination of Available Alkali in Pozzolans."

The work was conducted by the Concrete Laboratory, WES, under the direction of Mr. Bryant Mather and under the supervision of Mr. R. V. Tye, Jr. This report was prepared by Mr. W. G. Miller.

PG E. D. Peixotto, CE, and COL G. H. Hilt, CE, were Directors of WES during the preparation and publication of the report. Mr. F. R. Brown was Technical Director.

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### Summary

The procedure specified in acceptance testing of pozzolans for the determination of the available alkali released by a pozzolan requires the lime-pozzolan paste to be stored for a period of 28 days prior to test. In order to investigate the possibility of reducing the storage time of the paste prior to test, six pozzolans were tested for available alkali release rate by subjecting the paste to three conditions of storage time and/or temperature. The three conditions were as follows:

- a. Condition 1, Standard Condition, 28 days at 38 C.
- b. Condition 2, seven days at 38 C.
- c. Condition 3, seven days at 65.5 C.

The results of these tests are as follows:

- a. The highest available alkali values for all pozzolans were obtained under Storage Condition 1.
- b. All pozzolans, except calcined tuff, produced higher values for Condition 3 than for Condition 2.
- c. All pozzolans, except calcined tuff, produced available alkali values for Condition 3 that were in excess of two-thirds of the values produced for Condition 1.

Results of this study indicate that if the available alkali value of a pozzolan is 1.0 percent or less when the paste is stored at 65.5 C for seven days, the maximum specification limit of 1.5 percent will not be exceeded when the pozzolan is evaluated by the present Standard Condition, 28 days at 38 C.

In order to reduce the storage time of the lime-pozzolan paste to seven days, storage Condition 3 with a maximum limit of 1.0 percent available alkali is recommended as an optional method for the acceptance testing of pozzolan.

## INVESTIGATION OF ACCELERATED METHODS FOR THE DETERMINATION OF AVAILABLE ALKALI IN POZZOLANS

### Background

1. The Corps of Engineers Standard Guide Specification for Concrete, CE 1401.01,<sup>1</sup> requires the following conditions for release for shipment and approval for use of tested pozzolans: "Release for shipment and approval for use will be based on compliance with seven-day lime-pozzolan strength requirements and other physical and chemical and formity requirements for which test can be completed by the time the seven-day lime-pozzolan strength test is completed. Release for shipment and approval for use on the above basis will be contingent on continuing compliance with the other requirements of the specifications." The applicable portion of CRD-C 263, Corps of Engineers Method for Sampling and Testing Pozzolan for Use in Portland Cement Concrete,<sup>2</sup> for the determination of available alkali requires 28 days storage of the lime-pozzolan paste. Under current shipping and construction practices, most tested pozzolan is incorporated in the project construction prior to the completion of the above test.

2. This investigation was conducted as part of the responsibility assigned to the Waterways Experiment Station (WES) for the technical surveillance of cement and pozzolan procurement acceptance testing function of the Corps of Engineers as outlined in ER 1110-1-8100.<sup>3</sup>

### Purpose and Scope of Investigation

3. The investigation reported herein was undertaken to determine the alkali release rate of pozzolans and was confined to the effect of

storage time and temperature conditions on the available alkali released by lime-pozzolan paste prepared with pozzolans meeting the requirements of CRD-C 262, Corps of Engineers Specifications for Pozzolan for Use in Portland Cement Concrete.<sup>2</sup>

4. This report describes the materials tested, test methods used, results obtained, and conclusions derived therefrom.

### Materials and Test Methods

#### Materials

5. Pozzolans. From the many pozzolans available for use, six (four natural and two fly ash) were selected and identified as follows:

- a. Volcanic cinders (Vol C)
- b. Calcined Keasey Shale (CK Sh)
- c. Calcined Diatomaceous Shale (CD Sh)
- d. Calcined Tuff (CT)
- e. Fly Ash (FA I)
- f. Fly Ash (FA II)

6. Each pozzolan was blended in a one-quart blender for four hours. Approximately 150 grams of each material was placed in a sealed jar and retained for chemical analysis and preparation of lime-pozzolan paste for determination of available alkali. The remaining portion was placed in a plastic bag, sealed, and retained for use in the preparation of physical test specimens.

7. Lime. The lime conformed with the requirements of ASTM Designation C6-49, "Standard Specifications for Normal Finishing Hydrated Lime."<sup>4</sup>

8. Cement. The cement conformed with the requirements of CRD-C 200, "Federal Specification Cement, Portland."<sup>2</sup>

#### Test Methods

9. Tests conducted on each pozzolan were as follows:

- a. Evaluation of each material for compliance with the requirements of CRD-C 262.<sup>2</sup>
- b. Determination of the total acid soluble alkali in accordance with the procedure outlined in CRD-C 209, Method 14C1.1 para 4.1.<sup>2</sup>
- c. Determination of water soluble alkali in accordance with the procedure outlined in ASTM C 114, "Chemical Analysis of Hydraulic Cement."<sup>5</sup>
- d. The available alkali of lime-pozzolan paste was determined in accordance with the procedure outlined in CRD-C 263. Three rounds of test were conducted, a round consisting of the determination of the available alkali of each lime-pozzolan paste after exposure to the following storage conditions:

Condition 1, 28 days @ 38 C (Standard Condition)  
Condition 2, 7 days @ 38 C  
Condition 3, 7 days @ 65.5 C

#### Presentation and Analyses of Test Results

10. The results of tests for compliance with CRD-C 262, acid soluble alkali,\* and water soluble alkali are shown in table 1.

- a. All the pozzolans, except the CK Sh and CT, met the requirements of CRD-C 262. The CK Sh and CT failed to comply with the minimum 75 percent of control for pozzolanic strength of cement-pozzolan mortar at 28 days.

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\* The method used was that used for cement. In order to determine the total alkali content of a pozzolan, the material must be rendered soluble by fusion with ammonium chloride and calcium carbonate or some other appropriate material.

progress of the pozzolanic reaction or the additional exchange of calcium for potassium and sodium. At 65.5 C storage temperature the diffusion rates are more rapid and this is particularly true for the monovalent cations which have larger diffusion rates. Thus if the early calcium for potassium and sodium exchange takes place, the reverse exchange takes place to a more complete extent and less water-soluble alkali is available at seven days.

### Conclusions

13. Most pozzolans that produce an available alkali content of less than 1.0 percent when stored under Condition 3 would be expected to meet the specification requirement of 1.5 percent maximum when stored under Condition 1. Method 3 allows the completion of the test for available alkali in seven days and is recommended as an optional method in the acceptance testing of fly ash pozzolans. Method 3 is also recommended as an optional method for testing natural pozzolans which have been shown not to contain a zeolite.

### Literature Cited

1. Department of the Army, Office, Chief of Engineers, "Standard Guide Specifications for Concrete," CE 1401.01.
2. U. S. Army Engineer Waterways Experiment Station, CE, Handbook for Concrete and Cement, August 1949 (with Quarterly Supplements), Vicksburg, Miss.
3. Department of the Army, Office, Chief of Engineers, ER 1110-1-3100, "Laboratory Investigation and Material Testing for Military and Civil Works Construction Projects."
4. ASTM, "Standard Specification for Normal Finishing Hydrated Lime," Designation C6-49, 1968 Book of ASTM Standards, Part 9, 1968, Philadelphia, Pa.
5. ASTM, "Chemical Analysis of Hydraulic Cement," Designation C 114, 1968 Book of ASTM Standards, Part 9, 1968, Philadelphia, Pa.

TABLE 1

Results of Chemical Tests of Pozzolans, Percent

| <u>Sample</u> | <u>Loss on<br/>Ignition</u> | <u>Moisture</u> | <u>SO<sub>3</sub></u> | <u>SiO<sub>2</sub></u> | <u>R<sub>2</sub>O<sub>3</sub></u> | <u>SiO<sub>2</sub> +<br/>R<sub>2</sub>O<sub>3</sub></u> | <u>CaO</u> | <u>MgO</u> | <u>Acid Sol. Alkali</u>             |                        |                       | <u>Water<br/>Sol. Alk.<br/>Total<br/>as Na<sub>2</sub>O</u> |
|---------------|-----------------------------|-----------------|-----------------------|------------------------|-----------------------------------|---|------------|------------|-------------------------------------|------------------------|-----------------------|---|
|               |                             |                 |                       |                        |                                   |   |            |            | <u>Total<br/>as Na<sub>2</sub>O</u> | <u>Na<sub>2</sub>O</u> | <u>K<sub>2</sub>O</u> |   |
| Vol. C        | 1.69                        | 0.29            | 0.38                  | 54.2                   | 22.6                              | 76.8  | 9.9        | 3.1        | 0.52                                | 0.43                   | 0.14                  | 0.016   |
| CK Sh         | 1.28                        | 0.62            | 0.17                  | 61.7                   | 26.2                              | 87.9  | 5.0        | 2.9        | 0.42                                | 0.25                   | 0.26                  | 0.019   |
| CD Sh         | 1.76                        | 0.33            | 2.88                  | 60.1                   | 22.9                              | 83.0  | 8.1        | 2.5        | 0.32                                | 0.17                   | 0.23                  | 0.009   |
| CT            | 2.11                        | 1.99            | 0.04                  | 71.3                   | 16.2                              | 87.5  | 3.1        | 0.2        | 1.89                                | 0.44                   | 2.20                  | 0.032   |
| FA I          | 3.56                        | 0.13            | 0.85                  | 51.3                   | 35.6                              | 86.9  | 2.9        | 1.3        | 0.17                                | 0.07                   | 0.15                  | 0.012   |
| FA II         | 1.38                        | 0.13            | 2.02                  | 49.3                   | 38.4                              | 87.7  | 7.3        | 1.1        | 0.45                                | 0.28                   | 0.26                  | 0.063   |

TABLE 2

Results of Physical Tests of Pozzolans

| <u>Sample</u> | <u>Specific Gravity</u> | <u>Fineness Sq cm/cc</u> | <u>Water-Req. Increase in Flow %</u> | <u>Lime-Pozz Strength, psi*</u> | <u>Soundness Autoclave Exp., %</u> | <u>Pozz Activity % of Control</u> | <u>Incr. in Drying Shrinkage</u> |
|---------------|-------------------------|--------------------------|--------------------------------------|---------------------------------|------------------------------------|-----------------------------------|----------------------------------|
| Vol C         | 2.71                    | 20,135                   | 32                                   | 1095                            | -0.01                              | 86                                | 0.004                            |
| CK Sh         | 2.52                    | 24,075                   | 3                                    | 1300                            | -0.02                              | 74**                              | 0.006                            |
| CD Sh         | 2.61                    | 29,825                   | 17                                   | 1830                            | -0.03                              | 87                                | 0.008                            |
| CT            | 2.34                    | 28,190                   | 8                                    | 930                             | 0.02                               | 73**                              | 0.018                            |
| FA I          | 2.23                    | 8,240                    | 21                                   | 1330                            | 0.01                               | 94                                |                                  |
| FA II         | 2.52                    | 7,935                    | 23                                   | 1155                            | 0.01                               | 92                                |                                  |

\* psi x 0.006894757 = MPa

\*\* Failed to meet the minimum requirement of 75 percent.



TABLE 3

Results of Tests for Available Alkali of Pozzolans

| Sample<br>Symbol | Storage Conditions of Lime-Pozzolan Paste |           |                |           |                  |           |
|------------------|---|-----------|----------------|-----------|------------------|-----------|
|                  | Condition #1                              |           | Condition #2   |           | Condition #3     |           |
|                  | 28 days at 38 C                           |           | 7 days at 38 C |           | 7 days at 65.5 C |           |
|                  | Av. %                                     | Std. Dev. | Av. %          | Std. Dev. | Av. %            | Std. Dev. |
| Vol C            | 1.43                                      | 0.065     | 0.64           | 0.053     | 1.01             | 0.040     |
| CK Sh            | 0.57                                      | 0.035     | 0.29           | 0.047     | 0.48             | 0.029     |
| CD Sh            | 0.73                                      | 0.035     | 0.48           | 0.024     | 0.67             | 0.018     |
| CT               | 0.93                                      | 0.024     | 0.36           | 0.044     | 0.48             | 0.019     |
| FA I             | 0.81                                      | 0.120     | 0.11           | 0.012     | 0.76             | 0.035     |
| FA II            | 1.13                                      | 0.071     | 0.27           | 0.024     | 0.80             | 0.012     |

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